

APPENDIX 2. AUTOMATED SOLID-PHASE EXTRACTION PROCEDURE USING MIL-LILAB 1A WORKSTATION

Millilab 1A Solid-Phase Extraction Procedure
[mL, millileters; mL/min: milliliters per minute]

Estimated time for samples : 11 hours

Date : December 1, 1998

Tube name	Tube type
sample	PORT
elution	TUBE
splspike	TUBE
washprobe	TUBE
methelute	TUBE
Organic_waste	TUBE

Element name	Element type
Seppak+	CARTRIDGE

Port name	Liquid name
Syr1v1m1	sample
Syr1v1m2	sample
Syr1v1m3	sample
Syr1v1m4	sample
Syr1v2m1	sample
Syr1v2m2	sample
Syr1v2m3	sample

Syr1v2m4 sample

Syr1v3m1 sample

Syr1v3m2 sample

Syr1v3m3 sample

Syr1v3m4 sample

Syr1v4m1 sample

Syr1v4m2 sample

Syr1v4m3 distilled1

Syr2v1 distilled2

Syr2v2 ethyl acetate

Syr2v3 methanol

Pump name Syringe size

Pump 1 5.0 mL

Pump 2 1.0 mL

Reagent name Liquid name

Reagent_1 methanol

Reagent_2 d10-phenanthrene

Reagent_3 Not used

Reagent_4 ethyl acetate

Loop size

4 mL

Technique name

Technique parameters

(1) WASH PROBE Solvent = distilled1 Fill_Rate = 60 mL/min
 Empty_Rate = 60 mL/min Volume = 20 mL
 Strokes = 4

(2) SPE SELECT Cartridge = Seppak+

Technique name	Technique parameters
(3) SPE LOAD	Working_solvent = distilled1 Rate = 30 mL/min Empty_rate = 20 mL/min Volume = 1 mL Level = 0 From = methanol To = Organic_waste Gap = 0.1 mL
(4) SPE LOAD	Working_solvent = distilled1 Rate = 30 mL/min Empty_rate = 20 mL/min Volume = 1 mL Level = 0 From = ethyl acetate To = Organic_waste Gap = 0.1 mL
(5) SPE LOAD	Working_solvent = distilled1 Rate = 30 mL/min Empty_rate = 20 mL/min Volume = 1 mL Level = 0 From = methanol To = Organic_waste Gap = 0.1 mL
(6) SPE WASH	Solvent = distilled1 Rate = 30 mL/min Empty_rate = 20 mL/min Volume = 3 mL To = Waste
(7) WASH PROBE	Solvent = sample Fill Rate = 60 mL/min Empty_rate = 60 mL/min Volume = 15 mL Strokes = 3
(8) SPE WASH	Solvent = sample Rate = 30 mL/min Empty_rate = 20 mL/min Volume = 100 mL To = Waste

Technique name	Technique parameters
(9) WASH PROBE	Solvent = distilled1 Fill Rate = 60 mL/min Empty_rate = 60 mL/min Volume = 10 mL Strokes = 2
(10) ELEMENT PURGE	Element = Seppak+ Dispose = No Gas = Purge 6 Level = 0 Clear_time = 0.2 min Purge_time = 1 min To = Organic_waste
(11) SPE LOAD	Working_solvent = ethyl acetate Rate = 4.0 mL/min Empty_rate = 4.0 mL/min Volume = 3.5 mL Level = 0 From = ethyl acetate To = elution Gap = 0.1 mL
(12) GAS PURGE	Gas = Purge 6 To = Organic_waste Level = 900 Clear_time = 0 min Purge_time = 0.4 min
(13) ELEMENT PURGE	Element = Seppak+ Dispose = No Gas = Purge 6 Level = 0 Clear_time = 0 min Purge_time = 0.3 min To = elution
(14) SPE LOAD	Working_solvent = ethyl acetate Rate = 4.0 mL/min Empty_rate = 4.0 mL/min Volume = 3.5 mL Level = 0 From = methanol To = methelut Gap = 0.1 mL

Technique name	Technique parameters
(15) BATCH+PIPETTE	Working_solvent = ethyl acetate Fill_rate = 4 mL/min Empty_Rate = 4 mL/min Asperate_level = 60 Dispense_level = 560 Volume = 0.5 mL Gap = 0.1 mL From = d10-phenan To = elution Sample_count = All
(16) WASH PROBE	Solvent = ethyl acetate Fill_Rate = 6.0 mL/min Empty_Rate = 6.0 mL/min Volume = 2 mL Strokes = 4
(17) MIX	Working_solvent = ethyl acetate Fill_Rate = 6.0 mL/min Empty_Rate = 6.0 mL/min Asperate_level = 250 Dispense_level = 300 Volume = 2.5 mL Gap = 0.1 mL Count = 2 To = elution
(18) WASH PROBE	Solvent = ethyl acetate Fill_Rate = 6.0 mL/min Empty_Rate = 6.0 mL/min Volume = 2 mL Strokes = 4
(19) PIPETTE	Working_solvent = ethyl acetate Fill_Rate = 4.0 mL/min Empty_Rate = 4.0 mL/min Asperate_level = 270 Dispense_level = 550 Volume = 4 mL Gap = 0.2 mL From = elution To = Splspike

Technique name**Technique parameters**

- (20) MIX Working_solvent = ethyl acetate
Fill_Rate = 6.0 mL/min Empty_Rate = 6.0 mL/min
Asperate_level = 150 Dispense_level = 150
Volume = 3 mL Gap = 0.1 mL Count = 1
To = Washprobe
- (21) GAS PURGE Gas = Purge 6 To = Organic_waste Level = 900
Clear_time = 0 min Purge_time = 0.3 min
- (22) BUBBLE MIX Gas = Purge 6 To = Washprobe Level = 0
Clear_time = 0 min Purge_time = 0.3 min
- (23) WASH PROBE Solvent = ethyl acetate Fill_Rate = 6.0 mL/min
Empty_Rate = 6.0 mL/min Volume = 1 mL
Strokes = 2
- (24) SPE DONE Dispose = No